



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 8**

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Ref: 8EPR-SR

February 1, 2010

**MEMORANDUM**

**SUBJECT:** Proposed Pilot Study to Evaluate the Proficiency of the ESAT Soil Preparation Laboratory in Preparing Samples for Libby OU3 for Asbestos Analysis by PLM-VE

**FROM:** Bonnie Lavelle  
Remedial Project Manager  
Libby Asbestos Site, OU3

A handwritten signature in black ink, appearing to read "Bonnie Lavelle", is written over the typed name and title.

**TO:** Site File

The attached draft pilot study was prepared by SRC, Inc., and EPA contractor at the request of EPA. The purpose of the pilot study is to evaluate the proficiency of the ESAT soil preparation laboratory in preparing soil and soil-like samples collected from Libby OU3. During the Phase I remedial investigation of OU3, the soil preparation laboratory located in Troy, MT and operated by ESAT could not successfully prepare OU3 samples. There was an unacceptable loss of mass of the sample during the grinding operations. Once this problem was discovered, all OU3 samples were directed to the soil preparation facility in Denver, CO operated by CDM. The OU3 samples were successfully prepared at that facility and all Libby OU3 soil and soil-like samples designated for PLM-VE analysis have since been prepared at the CDM facility.

In 2009, the Libby Team leader directed CDM to dismantle the Denver facility. He further directed that all Libby soil and soil-like material were to be prepared for PLM analysis at a new laboratory being built in Libby, MT and operated by ESAT. Due to the historical problems with the ESAT grinders, this pilot study was deemed necessary to demonstrate that OU3 samples could be successfully ground and prepared at the new facility in Libby.

Through an Interagency Agreement with the Department of Transportation, I was able to arrange for OU3 samples to continue to be prepared at the Denver facility by CDM.

The attached pilot study was thus never finalized. If, in the future, it becomes necessary to use the ESAT soil preparation laboratory for OU3 samples, this pilot study should be finalized and implemented before OU3 field samples are sent. I recommend that in addition to the samples identified by SRC, Inc. in the attached study, additional sediment samples be included to increase the total number of samples to 20.

Attachment

**SOIL PREPARATION PILOT STUDY  
LIBBY ASBESTOS SUPERFUND SITE  
OPERABLE UNIT 3**

## **1.0 INTRODUCTION**

The U.S. Environmental Protection Agency (EPA) has developed an analytical method for estimating the concentration of Libby Amphibole (LA) asbestos in samples of soil and soil-like materials collected from the Libby Asbestos Superfund site. The most recent version of the SOP for this method (ISSI-LIBBY-01 Rev. 10) is provided as Attachment 1.

In brief, a soil or soil-like sample is prepared for analysis by grinding in a plate grinder to produce particles that are  $\leq 250$   $\mu\text{m}$  in diameter. Following grinding, the material is examined by polarized light microscopy (PLM) using two standard reference materials (0.2% and 1.0% LA by mass) to provide a semi-quantitative estimate of the LA level in the test material. Possible categories are as follows:

Category	Meaning
Bin A	LA was not detected in the sample
Bin B1	LA was detected at a level that is less than the 0.2% reference material
Bin B2	LA was detected at a level equal to or greater than the 0.2% reference material, but at a level less than the 1% reference material
Bin C	LA was detected at a level equal to or greater than the 1% reference material

Until recently, soil and other soil-like samples from the Libby site, including some samples from Operable Unit 3 (OU3), were prepared for PLM analysis at a laboratory in Denver that was operated on behalf of EPA by CDM Federal Services. More recently, EPA has determined that responsibility for preparation of soil samples from the Libby site will be shifted to a laboratory in Libby, operated by EPA's Environmental Services Assistance Team (ESAT).

The purpose of this Pilot Study is to describe a limited series of checks to ensure that preparation of soil and soil-like materials from OU3 by the ESAT laboratory will yield samples that are suitable for analysis by PLM, and will be comparable to the preparation of previous soil and soil-like samples from OU3.

## 2.0 PROFICIENCY DEMONSTRATION

### 2.1 Performance Requirements

The SOP for soil preparation (Attachment 1) identifies a number of requirements that must be satisfied in order for the soil preparation process to be considered acceptable. This includes the following:

- a. All of the material from the grinder must pass through a 250 um (60-mesh) screen, and must be largely retained on a 74 um (200-mesh) screen (see Section 9.1 of the SOP)
- b. The mass of material recovered from the grinder must be no less than 90% of the mass of material placed into the grinder (see Section 9.3 of the SOP)
- c. All preparation blanks and grinding blanks must be non-detect (Bin A) for LA when analyzed by PLM (see SOP Section 12.1 and 12.2).
- d. The frequency of strongly discordant results for duplicate preparations must not exceed 10% (see SOP Section 12.4)

These requirements of the SOP form the basis for the pilot study to evaluate the soil preparation performance of the ESAT facility.

### 2.2 Study Design

In order to determine if the requirements specified above are satisfied, a set of 10 soil samples previously collected from OU3 will be provided to ESAT for preparation. These 10 samples will be obtained from archived splits of unprocessed samples that have been previously collected at OU3. **If the sample mass is insufficient, backup samples have been selected.**

ESAT will prepare these 10 samples for PLM analysis in accord with SOP ISSI-LIBBY-01 (Revision 10), along with two grinding blanks and two preparation blanks, as shown in Table 1.

### 2.3 Sample Preparation Data Collection by ESAT

For each of the 14 samples, ESAT will record the mass of the sample placed into the plate grinder, and the mass of ground material recovered from the grinder.

For each of the 14 samples, ESAT will sieve the ground material through nested 60 mesh and 200 mesh screens and report the mass retained on each screen.

Table 2 is a template for data recording for this phase of the pilot study.

## 2.4 PLM Analysis

PLM analyses of prepared samples will be performed by the same laboratory that performed the original PLM analysis. This is to minimize any bias that might arise from inter-laboratory variations in PLM analysis techniques.

The first samples to be analyzed will be the two grinding blanks and the two preparation blanks.

If these 4 samples are all reported to be ND (Bin A), then analysis of the 10 remaining OU3 samples will be performed. As discussed in the SOP, due to the subjective nature of the PLM analysis and to random variation between different splits of soil, it is not expected that all PLM reanalyses will be identical to the original results. However, it is required that the frequency of strongly discordant results (differing by two or more bins) not exceed 10%.

## 2.5 Data Evaluation

In accord with the requirements of the SOP, soil preparation will be considered acceptable if the following conditions are satisfied:

- Mass recovery is at least 90% for all samples
- At least [REDACTED] of the mass is retained on the 200 mesh screen for all samples
- All preparation blanks and grinding blanks are ND (Bin A)
- The frequency of strongly discordant results is 10% or less

**TABLE 1**  
**SAMPLES FOR PREPARATION**

Sample	Description
1	OU3-P1-00130
2	OU3-P1-00132
3	Preparation blank
4	OU3-P1-00140
5	OU3-P1-00145
6	Grinding blank
7	OU3-P1-00164
8	OU3-P1-00206
9	Preparation blank
10	OU3-P1-00226
11	OU3-P1-00344
12	Grinding blank
13	OU3-P1-00365
14	OU3-P1-00378

**TABLE 2**  
**DATA RECORDING TEMPLATE**

Sample	Orig Mass (g)	Mass Recovered (g)	% Recovered	Mass retained on 60 mesh (g)	Mass retained on 200 mesh (g)	% In target size range (a)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						

(a) % in target size range = Mass on 200 mesh screen / Mass Recovered

IndexID	Media	AnalysisLab	MFLA%	Fine Fraction (g)	Lab Sample ID
P1-00130	Forest Soil	EMSL27	A	177	270800023-270800023-0005
P1-00132	Forest Soil	EMSL27	A	178.6	270800021-270800021-0008
P1-00140	Forest Soil	EMSL27	6	274.4	270800017-270800017-0009
P1-00145	Forest Soil	EMSL27	A	176.5	270800029-270800029-0003
P1-00164	Forest Soil	EMSL27	B1	136.6	270800021-270800021-0002
P1-00206	Mine Waste	EMSL27	5	142.7	270800014-270800014-0032
P1-00226	Forest Soil	EMSL27	B1	122.8	270800011-270800011-0047
P1-00344	Mine Waste	EMSL27	B2	179.8	270800011-270800011-0001
P1-00365	Mine Waste	EMSL27	B1	154.7	270800011-270800011-0016
P1-00378	Mine Waste	EMSL27	B2	183.6	270800014-270800014-0046

Extra samples

P1-00205	Mine Waste	EMSL27	2	192.5	270800014-270800014-0031
P1-00152	Forest Soil	EMSL27	A	197.8	270800016-270800016-0002
P1-00372	Mine Waste	EMSL27	B1	164.8	270800011-270800011-0014
P1-00389	Mine Waste	EMSL27	B2	187.4	270800014-270800014-0051

*NOTE: This SOP has been prepared for use at the Libby Asbestos Superfund Site. The applicability of this SOP at other sites should be evaluated by the site team with regard to site-specific goals and objectives.*

Date: December 6, 2007

SOP No. ISSI-LIBBY-01 (Rev. 10)

Title: SOIL SAMPLE PREPARATION

**SYNOPSIS:** A standardized method for preparation of soil samples for asbestos analysis at the Libby Asbestos Superfund Site is described.

Original Author: William Brattin

Syracuse Research Corporation<sup>1</sup>

Received by QA Unit:

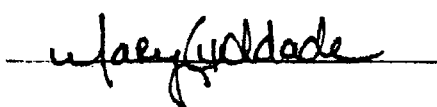
**APPROVALS:**

**TEAM MEMBER**

**SIGNATURE/TITLE**

**DATE**

EPA Region 8:



12/11/07

Syracuse Research Corp.



12/10/07

<sup>1</sup> This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by Syracuse Research Corporation (SRC).



### REVISION LOG

Revision Number	Revision Date	Reason for Revision
1	1/7/00	Incorporation of sieving to the sample preparation.
2	7/12/00	Revision in sieve size, other minor edits.
3	5/7/02	Incorporate minor edits
4	8/1/02	Modify sieving procedure, add grinding step
5	3/6/03	Incorporate modifications to the procedure and documentation requirements
6	3/24/03	Incorporate modifications to the log-sheets to conform with electronic data storage requirements and add grinder blank requirements.
7	8/5/03	Incorporate modifications to drying and sample storage procedures
8	5/4/04	Incorporate modifications to drying batch size and recording of preparation information
9	5/14/07	<p>Incorporate modifications so as to expand use to other Operable Units (removed references to OU4 / CSF, changed Index ID to Sample ID). Repair formatting. Remove reference to missing Figure 1. Add optional use of electronic logs. Oven temperature set to <math>90 \pm 10</math> degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designate drying batch as one batch per oven (~20 samples). Allow for optional use of disposable drying pans. Remove direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily.</p> <p><i>[Note: Revision 9 was an unsigned version that reflects changes made at the Troy Preparation Laboratory. Some of the changes in Revision 9 are retained in Revision 10, below].</i></p>
10	12/06/07	<p>Incorporate modifications so as to expand use to other Operable Units. Designate drying batch as ~20 samples. Allow for optional use of disposable drying pans. Allow alternative methods for decontamination of plate grinder. Clarify and modify QC requirements. General editing for clarity.</p>

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) has been prepared by the United States Environmental Protection Agency (USEPA) Region 8 to standardize the methods used to prepare soil samples from the Libby Asbestos Superfund Site for the analysis of asbestos content. This procedure is intended for use by employees of USEPA Region 8 and by contractors and subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby site. Deviations from the procedures outlined in this document must be reviewed and approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist.

## **2.0 RESPONSIBILITIES**

Each laboratory that performs soil preparation activities under this SOP must have a designated Preparation Laboratory Project Leader (PL<sup>2</sup>). The PL<sup>2</sup> may be an USEPA employee or contractor. The PL<sup>2</sup> is responsible for ensuring that all personnel in the laboratory who perform work under this SOP are familiar with the SOP, and for ensuring that all work performed satisfies the requirements of this SOP and any other relevant laboratory-specific operating procedures. It is also the responsibility of the PL<sup>2</sup> to communicate and document the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

All laboratory personnel preparing Libby soil samples are responsible for reading and understanding the requirement of this SOP, and for performing all applicable tasks in accordance with this SOP. Any laboratory worker who identifies any issues or encounters any difficulties in implementation of this SOP is responsible for promptly communicating the issue or difficulty to the PL<sup>2</sup>. In addition, all laboratory personnel are responsible for reading and understanding the Health and Safety Plan (HASP) applicable to the soil preparation activities in that laboratory, and performing all tasks in accord with the requirements of that HASP.

## **3.0 EQUIPMENT**

- General purpose laboratory oven - capable of maintaining a constant temperature of approximately 90°C.
- Analytical balance - capable of measuring in a range of 0.1 g to at least 2000 g, calibrated and accurate to the tolerance limits indicated in Attachment 2.
- Riffle splitter - with 3/4 inch chutes to split samples.

- Plate grinder - capable of accepting soil particles of approximately 1/4 inch diameter and grinding to produce particles of approximately 250  $\mu\text{m}$ .
- HEPA Vacuum - A portable vacuum unit equipped with a high efficiency particulate air (HEPA) filter to remove any asbestos fibers and other soil particles from the exhaust air. Used to decontaminate equipment and maintain general laboratory cleanliness.
- Metal scoop or spoon - for transferring samples. Plastic scoops or spoons are not acceptable.
- 1/4 inch metal sieve and catch pan - for coarse sieving samples. Plastic sieves and pans are not acceptable.
- 60 mesh (250  $\mu\text{m}$ ) and 200 mesh (74  $\mu\text{m}$ ) metal sieves - for verification of the plate grinder settings. Plastic sieves are not acceptable.
- Clean quartz sand - required for preparation of grinding and drying blank samples and for decontamination of grinder.
- Clean soil - required for calibration of grinder.
- Drying pans with lids - used during the sample drying process, lids used to cover samples during transfer
- Sample containers - plastic ziplock bags (pint and gallon size).
- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.
- Personal Protective Equipment - as specified in the applicable Health and Safety Plan for the soil preparation laboratory.
- Laboratory notebook and pen - used to record progress, any problems or observations and deviations. All information in the laboratory notebook must be recorded in pen (not pencil).
- Sample Drying Log Sheets - (Attachment 1). Used to record all sample drying information.

- Sample Preparation Log Sheets - (Attachment 1). Used to record all sample preparation information (splitting, sieving and grinding).
- Equipment Calibration and Maintenance Logs for:
  - Analytical Balance (Attachment 2)
  - Plate Grinder (Attachment 3)
  - Ventilation Hood (Attachment 4)
  - HEPA Vacuum (Attachment 5)
  - Drying Oven (Attachment 6)

These logs are used to record all maintenance and calibration records for the listed equipment. If hard copy, all entries must be recorded in pen, and the logs must be organized and maintained in a laboratory notebook.

- Sample Labels – Self-adhesive labels for attachment to sample bags.
- Trash Bags - used to dispose of gloves, wipes and other investigation derived waste.
- Indelible Marking Pen - used to record sample information onto plastic ziplock bags and to record logbook information.

#### **4.0 METHOD SUMMARY**

Figure 1 provides an overview of the steps in the soil preparation process. Soil samples received from the field are first dried in a laboratory oven and are then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material ( $> 1/4$  inch) from fine material ( $< 1/4$  inch). The fine material is ground to a particle size of less than 250  $\mu\text{m}$ , and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for microscopic analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an analytical laboratory for asbestos analysis by methods specified in the project-specific Sampling and Analysis Plan. At present, the fine-ground sample is generally analyzed by Phase Contrast Microscopy (Visual Area Estimation) (PLM-VE) in accord with the most recent version of SOP SRC-LIBBY-03, and the coarse material is examined by stereomicroscopy and any observable particles of asbestos are removed and weighted in accord with the most recent version of SOP SRC-LIBBY-01.

It should be noted that this preparation method, coupled with these analytical techniques, is intended to estimate the total mass fraction of asbestos that is present in a sample, without regard

to the current size distribution of the asbestos particles. That is, no distinction is drawn between asbestos that is presently in a large "lump" that is non-respirable and free asbestos fibers that are readily released to air and inhaled. Because of this, concentration values based on this approach may tend to overestimate the amount of currently releasable fibers, but do provide an estimate of the total amount of fibers that may be releasable in the future.

## **5.0 SOIL STORAGE**

Upon receipt at the soil preparation facility, samples will be grouped into an inventory batch of 50-120 samples. Samples will be archived according to the inventory batch they are assigned to and filed by the Inventory Batch ID (box number) noted in the Sample Drying Log and Sample Preparation Log (Attachment 1).

## **6.0 BULK SOIL DRYING**

### **6.1 Equipment Calibration**

Samples will be weighed prior to and following drying activities. The analytical balance used for drying activities will be calibrated on days when samples are loaded into, or unloaded from, the oven. Before weighing samples, calibrate the balance using S-1 class weights and record all measurements, any required maintenance, and the balance number in the Analytical Balance Calibration and Maintenance Log (Attachment 2).

All drying activities will be performed under a negative pressure HEPA filtered hood or similar containment box. Prior to loading the oven, the ventilation hood will be calibrated to ensure that the ventilation system is operating properly. Ventilation hood calibration and any required maintenance will be documented in the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. Vacuum calibration will be performed daily, prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance Log (Attachment 5).

Oven temperature calibration will be performed on a daily basis (during periods of operation). Oven temperature calibration and any required maintenance will be documented in the Oven Temperature Calibration and Maintenance Log (Attachment 6).

## 6.2 Drying Procedure

- Prior to unsealing and drying each sample, record on the Sample Drying Log the starting sample mass to the nearest 0.1 g. Include the technicians initials and the date.
- Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying Log, along with the SOP and Revision Number and the oven number used to dry the samples.
- Include one preparation blank in each drying batch. See Section 12.1 for more details regarding preparation blanks.
- Set the oven temperature to approximately  $90 \pm 1^\circ\text{C}$ . For every drying batch, check the oven temperature to verify that proper temperature<sup>2</sup> has been reached and document the start date/time and temperature in the Sample Drying Log.
- Transfer each sample to be dried from its ziplock storage bag into a clean drying pan. Each sample should be transferred to its respective drying pan under the negative pressure HEPA filtered hood. Label each drying pan with the Index ID<sup>3</sup> of the sample. Place each sample in the oven.
- Leave the samples in the oven for approximately 24-48 hours or until completely dry. Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness. Once it is confirmed that samples are dry, record the technician's initials, and the date and time of completion, in the Sample Drying Log.
- Turn off the oven and allow the samples to cool in the oven. Once the samples are cooled, unload each sample and transfer each sample volume to a clean ziplock bag, re-bag the sample with another clean ziplock bag and identify the dried sample with the Index ID. All samples should be transferred to ziplock bags under the negative pressure HEPA filtered hood to prevent potential exposure to fibers that might be released from the sample.
- Record the sample mass of each dried and bagged sample to the nearest 0.1 g along with the technician's initials and the date in the Sample Drying Log.

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<sup>2</sup> Drying temperatures in the range of 80-100°C will not compromise sample integrity, but monitoring of oven temperature to  $\pm 1^\circ\text{C}$  is needed to allow early detection of any problems with the oven temperature control.

<sup>3</sup> Unique sample identifiers at the Libby site are referred to as "Index ID" numbers rather than "Sample ID" numbers. However, the meaning is the same.

### **6.3 Decontamination**

Decontaminate the inside of the hood and the inside of the drying oven by HEPA vacuuming and wet wiping all surfaces before loading a new batch for drying.

If drying pans are to be re-used, decontaminate all sample drying pans under the ventilation hood using compressed air and a HEPA vacuum to remove any residual organic material left on the pans. Wet wipe or brush off any visible material that is not removed using the vacuum.

## **7.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES**

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for asbestos analysis. The sections below describe the sample splitting procedure.

### **7.1 Equipment Calibration**

Prior to any splitting, sieving, or grinding activities, calibrate the ventilation hood to ensure that the ventilation system is operating properly. Document ventilation hood calibration and any required maintenance in the Ventilation Hood Calibration and Maintenance Log.

### **7.2 Procedure for Sample Splitting**

Splitting must be performed in the hood to prevent potential exposure to fibers that might be released from the sample. Samples will be divided using the following steps:

- Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.
- Place one collection pan on each side of the riffle splitter. Pour the sample from its plastic bag through the splitter in order to divide the sample into two equal sub-parts.
- After splitting, set aside one portion for sample preparation, as described below. If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that 3/4 of the original sample will be archived and 1/4 will be set aside for processing.
- Place the remaining portion(s) into a clean, ziplock bag, re-bag the sample in another clean ziplock bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Index ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation

Log. Store the archive portion in the numbered inventory box noted in the Sample Preparation Log.

### **7.3 Preparation Duplicate Samples**

One preparation duplicate sample will be prepared for every 20 field samples processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Index ID and both the parent sample and the duplicate sample are then processed in an identical fashion and are each submitted to the laboratory blind. For further information on preparation and processing of preparation duplicates, refer to Section 12.4.

### **7.4 Performance Evaluation Samples**

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 12.3.

### **7.5 Decontamination**

The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine ground fraction of the same samples into four fractions (see Section 10, below). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.

- Use a HEPA vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

## **8.0 SIEVING THE PREPARATION SAMPLE**

All preparation samples are sieved prior to grinding to separate out the coarse and fine fractions. The sample sieving procedure is described in the sections below.

### **8.1 Equipment Calibration**

All sieving activities will take place in the hood. Refer to Section 6.1 for details regarding the frequency of ventilation hood calibration.

Samples are weighed during sieving activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance,



and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

## **8.2 Sample Sieving Procedure**

Samples will be sieved using the procedure outlined below.

- Pour the sample onto a clean 1/4 inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <1/4 inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <1/4 in size pass through the screen.
- Pour all material which does not pass through the screen (>1/4 inch) into a new, tared, sample bag. This is the Coarse Fraction.
- Weigh and record the mass of the coarse fraction to the nearest 0.1 g in the Sample Preparation Log and record the technician's initials and the date. If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction in the Sample Preparation Log.
- Double-bag the coarse sample portion and identify the sample with the Index ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.
- All material that passes through the 1/4 inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g in the Sample Preparation Log.

Whenever possible, immediately process the fine fraction material in accord with the approach described in Section 9.3 (below). If processing cannot occur immediately, pour the fine fraction material into a new ziplock bag and identify the fine sample material with the Index ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Index ID and suffix on the outside of the bag.

## **8.3 Decontamination**

All non-disposable pans and sieves will be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) under the ventilation hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

## **9.0 GRINDING THE FINE FRACTION**

The fine fraction of each preparation sample will be ground to produce a material of about 250  $\mu\text{m}$ <sup>4</sup>. The procedure for grinding the fine fraction is outlined below.

### **9.1 Equipment Calibration**

All grinding activities will take place in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

A HEPA vacuum will be used to decontaminate the hood and processing equipment, following the preparation of each sample. Vacuum calibration will be performed daily, prior to grinding activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance.

A plate grinder will be used to process samples. The grinder will be calibrated daily or after any adjustments are made to the plates. To verify proper particle size (approximately 250  $\mu\text{m}$ ), and demonstrate that samples will not be over-processed, grind a sample of clean soil (rather than quartz sand) and sieve using stacked sieves. Clean soil will be provided by the United States Geological Survey (USGS). Unlike the coarseness of quartz sand, clean soil will more accurately approximate the typical grain size and texture of the Libby samples being processed and will reduce the chance of over-processing.

The grinder is adjusted acceptably if, after grinding of the clean soil sample, all material passes through a 60-mesh (250  $\mu\text{m}$ ) screen and is substantially retained by a 200-mesh (74  $\mu\text{m}$ ) sieve. If a significant amount of the ground clean soil sample is retained on the 60-mesh screen, or if a substantial fraction of the material passes through the 200-mesh screen, adjust the plates of the grinder until these targets are achieved. If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Regardless, grinding of field samples cannot resume until the desired particle size is achieved. Document the grinder number, verification of acceptable adjustment and any observations in the Grinder Calibration and Maintenance Log.

Samples will be weighed following grinding activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

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<sup>4</sup> Note that the particle size is cited as "approximately 250  $\mu\text{m}$ ". This is due to the nature of grinding asbestos material. Some material that is longer than 250  $\mu\text{m}$  may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250  $\mu\text{m}$

## **9.2 Grinding Blanks**

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 12.2.

## **9.3 Grinding of Fine Field Samples**

The sample portion that was sieved to < 1/4 inch will be ground to a particle size of approximately 250  $\mu\text{m}$ . Set up a catch pan under the grinder to collect all the ground material. Take the fine sample set aside in Section 8.2, load the grinder hopper, and allow the fine sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number in the Sample Preparation Log.

The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

## **9.4 Decontamination**

### *Plate Grinder*

The details of decontamination of the plate grinder and its associated containers and equipment may vary depending on the model of grinder that is being used.

If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, use wet wipes to ensure decontamination. If wet wipes are used, the plates and chutes must be thoroughly dried before reassembly. If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps). Then, pass an aliquot of approximately 20 g of quartz sand through the grinder to clean out any residual soil. Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated by using a HEPA vacuum and/or wet wipes, followed by a blast of high pressure air.

### *Calibration Sieves*

The stacked sieves used to calibrate the plate grinder will be decontaminated using a HEPA vacuum and compressed air between calibration uses.

## **10.0 SPLITTING OF THE FINE GROUND SAMPLE**

The fine ground soil sample should be distributed into four approximately equal subsamples using a splitter. All splitting activities will be performed in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

### **10.1 Splitting Procedure for Fine Ground Sample**

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997):

- Set up one receiving pan on each side of the splitter. Load the soil from the grinder catch pan (Section 9.3) into the splitter, collecting the sample in two receiving pans.
- Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.
- Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.
- Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above. Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions in the Sample Preparation Log.
- Take these two portions and carefully transfer each into a clean, tared, ziplock sample bag. Re-bag one sample portion in another clean ziplock sample bag and identify this fine ground sample with the Index ID, the suffix "FG" (for "fine fraction, ground") and the fraction number 1, (ex. CS-12345-FG1 for fine ground fraction #1). Identify the bagged second portion with the Index ID, the suffix "FG" and the fraction number 2 and set aside to be re-bagged with the following fine ground portions:

- Place the two empty receiving pans from the "FG1" and "FG2" portion next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3" and "FG4" portions.
- Take the remaining "FG3" and "FG4" portions and carefully transfer each into a clean, tared, ziplock sample bag, identify each remaining fine ground sample with the Index ID as noted above.
- Weigh each sample portion (FG1 through FG4), and record each mass along with the technician's initials and date in the Sample Preparation Log.

Combine all of the bagged coarse and fine portions of the sample into one large clean, ziplock sample bag.

Coarse and fine ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, the "FG1" fraction will be sent first. If further analyses are required for the fine ground portion, the subsequent fractions will be double bagged and sent (i.e., FG-2 then FG-3, etc.). All archived fine ground portions will be filed in the appropriate inventory archive box noted in the Sample Preparation Log.

## **10.2 Decontamination**

The splitter must be decontaminated between each sample. Use the vacuum and/or wet wipes followed by a blast of compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

## **11.0 DOCUMENTATION**

Index ID numbers are recorded in the Sample Drying Log, Sample Preparation Log and on all sample containers. Sample Drying Logs and Sample Preparation Logs will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying Log and/or Sample Preparation Log are necessary, the appropriate parties will be notified of the changes, however, these changes will not necessitate revision to the current standard operating procedure, a modification form will be filled out to document the revisions.

As mentioned above, the following equipment calibration and maintenance logs will also be maintained:

- Daily analytical balance calibration using S-1 class weights (Attachment 2)
- Daily grinder setting verification for calibration check and/or post-adjustment verification, grinder maintenance as necessary (Attachment 3)
- Daily ventilation hood operating condition verification (i.e., inline filter checks, changes) (Attachment 4)
- HEPA vacuum maintenance and bag changes (Attachment 5)
- Weekly oven temperature calibration, oven maintenance as necessary (Attachment 6)

In addition, a laboratory notebook will be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected:

- Date
- Time
- Personnel
- Personal protective equipment (PPE)
- SOP (including revision number) and any other laboratory-specific governing plan being followed
- Descriptions of any deviations to the SOP, the reason for the deviation and/or any modification forms being followed
- Summary of laboratory activities (including number of samples prepared, and equipment calibrated and used)

## **12.0 QUALITY CONTROL**

Quality control (QC) samples are inserted into the sample train to monitor for potential contamination introduced during the preparation process or to assess accuracy of analysis that may be affected due to preparation procedures. If samples results indicate the occurrence of contamination or inconsistent results, the PL<sup>2</sup> will be notified. The PL<sup>2</sup> will then notify the EPA Regional Project Manager and the Regional Chemist in order to review laboratory procedures and identify any changes in preparation laboratory methods and procedures that may be necessary. Any such reviews and resultant changes will be documented accordingly by the PL<sup>2</sup>.

### **12.1 Preparation Blanks**

A preparation blank is a sample of 200-400 grams of clean quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼ inch screen (even though there are no particles larger than ¼ inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.

At least one preparation blank will be processed with each drying batch of approximately 20 field samples. Preparation blanks will be assigned a random and unique Index ID and will be submitted to the laboratory blind. The Index ID assigned to each preparation blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any preparation blanks at a level greater than Non-detect (Bin A) by PLM-VE should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos (> Bin A) exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

## **12.2 Grinding Blanks**

A grinding blank consists of 100-200 grams of clean quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.

One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation. The grinder number used for each sample will be noted in the Sample Preparation Log. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine ground samples. The grinding blank is assigned a random and unique Index ID and is submitted to the laboratory blind. The Index ID assigned to each grinding blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any grinding blank at a level greater than Non-detect (Bin A) should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos (> Bin A) in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

## **12.3 Performance Evaluation Samples**

Performance Evaluation (PE) samples are samples of Libby soil that have been spiked with a known amount of Libby Amphibole (LA) asbestos. These samples were prepared by the USGS

for use at the Libby site by spiking uncontaminated soil from Libby with a known mass of LA fibers collected at the mine site, and then grinding the sample to a particle size of  $\leq 250$   $\mu\text{m}$  as described above. Several different concentration values of PE samples were prepared, ranging from  $< 0.1\%$  to  $2\%$ . Each bottle contains about 100 grams of the PE material.

PE samples will be utilized in two ways.

First, the soil preparation facility will insert untreated PE samples into the analytical sample train sent to the laboratory for PLM-VE analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the preparation facility).

Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, as detailed below. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.

The frequency of each type of PE sample (unprocessed and processed) should be one per month for each month in which soil processing is occurring. These should be distributed approximately evenly between the different concentration values that are available for PE samples.

Each month that soil processing is occurring, the procedure to be followed for generation and submittal of PE samples is as follows:

1. Select a PE bottle for inclusion.
2. Thorough mix the contents of the PE bottle by inversion (a minimum of 10 times) and/or rolling (a minimum of 10 minutes).
3. Remove an aliquot of about 20 grams and package this for submission to the analytical laboratory without any processing. If more than one laboratory is analyzing samples, rotate the submittal of unprocessed samples so that all laboratories receive approximately equal total number of unprocessed PE samples.
4. Take the remainder of the PE bottle (about 80 grams) and carry this material through the full sequence of steps applied to each field sample, starting with oven drying. After splitting the dried sample with the riffle splitter, recombine the samples so that the full 80 grams is screened through the  $\frac{1}{4}$  inch sieve and passed through the plate grinder. Thus, there is no archive split for PE samples. After grinding and splitting, this should result in four sub-samples of processed PE sample. Prepare three of these for submittal to the analytical laboratories, and hold one sample in archive.



Results of PE samples processed by the soil preparation laboratory are evaluated by comparing the reported results for LA to the nominal results. Deviations from nominal may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results (i.e., the results of the PE sample differ by more than one bin from the nominal result) exceeds 10%, then the source of the inconstancy should be investigated and remedied.

#### **12.4 Preparation Duplicates**

A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Index ID, and is submitted to the laboratory blind. The Index ID assigned to each preparation duplicate must be in accord with the numbering system specified in the program-specific project plan.

One preparation duplicate sample will be processed for every 20 field samples prepared (5%). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate may be due either to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.

#### **13.0 DECONTAMINATION**

All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves and drying pans that are re-used must be decontaminated with a HEPA vacuum, compressed air, wet-wiping and/or by brushing off any residual material. If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean. To reduce the potential for human exposure in the laboratory, COMPRESSED AIR SHOULD BE USED CAREFULLY AND ONLY UNDER VENTED HOODS.

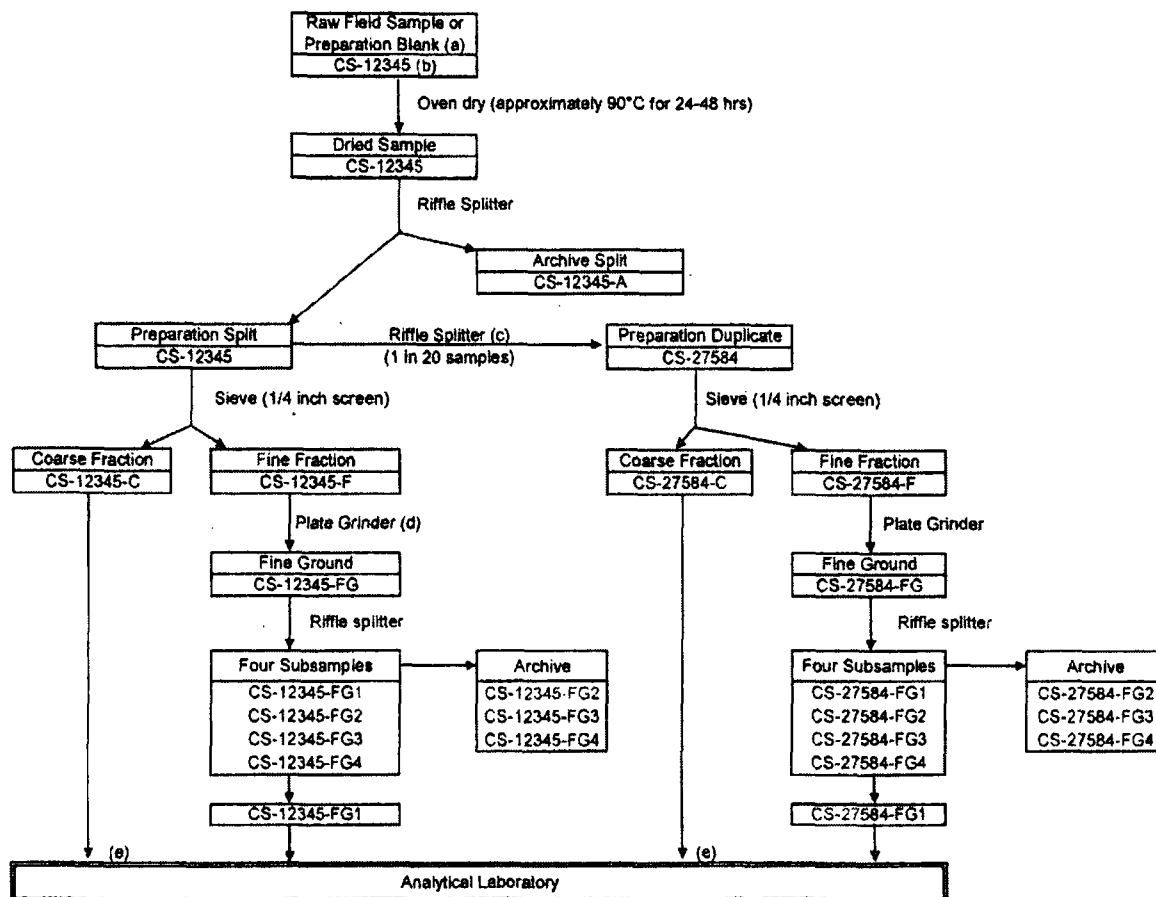
Detailed decontamination procedures for specific equipment are noted in Sections 6.3, 7.5, 8.3, 9.4, and 10.2.

#### **14.0 REFERENCES**

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

USEPA. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

FIGURE 1 SOIL PREPARATION FLOW DIAGRAM



NOTES:

- (a) A preparation blank (200-400 grams of clean silica sand) is prepared in the same way as field samples at a rate of 5%
- (b) Example index ID (sample number) shown to illustrate naming conventions
- (c) A preparation duplicate is prepared at a rate of 5%
- (d) A grinding blank (100-200 grams of clean sand) is passed through the plate grinder and split into 4 sub-samples at a rate of 5%
- (e) Coarse sample will be returned to EPA for archive after laboratory analysis

**ATTACHMENT 1**

**SAMPLE DRYING AND SAMPLE PREPARATION LOG SHEETS**

## Sample Drying Log Sheet

Laboratory Name: \_\_\_\_\_

Sheet No.: \_\_\_\_\_

Drying Begun: date \_\_\_\_\_ time \_\_\_\_\_

Drying Complete: date \_\_\_\_\_ time \_\_\_\_\_

Oven number: \_\_\_\_\_

Oven temp: \_\_\_\_\_ °C

	Index ID	Inventory ID No.	SOP and Rev No.	Sample mass (g)			Original Sample ID and Notes (indicate if preparation blank)	QC Initials and Date
				Before drying	After Drying	Initials and date		
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

## Sample Preparation Log Sheet

Laboratory Name: \_\_\_\_\_  
 Preparation Batch: \_\_\_\_\_

Sheet No.: \_\_\_\_\_

Index ID	SOP and Rev No.	Inventory ID	Drying Batch ID	Archive Sample Splitting Initials and date	Duplicate Sample Splitting Initials and date	Sieving		Initials and Date	Sample Grinding		Sample Splitting					Initials and Date	Original Sample Identification and Notes (Indicate if grind blank, prep blank, or duplicate pair. For duplicate pair enter the parent ID)	QC Initials and Date
						Sample Mass (g) Coarse Fraction > 1/4"	Sample Mass (g) Fine Fraction < 1/4"		Initials and Date	Grinder #	Sample Mass (g)				Initials and Date			
											FG1	FG2	FG3	FG4				
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18																		
19																		
20																		

The following preparation steps require Technician Initials and Date to document activity. Sample Drying, Archive Sample Splitting, Preparation Duplicate Splitting, Sieving, Homogenization, Sample Splitting

ATTACHMENT 2

ANALYTICAL BALANCE CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = \_\_\_\_\_

Balance # = \_\_\_\_\_

Measurement Number	S - 1 Class Weight Measurements				Measurement within range? Yes or No	If "No" Recalibrate	Technician Initials	QC check initials	
	Calibration Weights	0.1 g	1 g	10 g					100 g
	Tolerance Limit Range	0.05 - 0.15 g	0.90 - 1.10 g	9.75 - 10.25 g					99.00 - 101.00 g
Date									
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									

The analytical balance calibration will be verified daily.  
 All tolerance limits are standard tolerance limits for Class S-1 weights.  
 After 20 measurements, the tolerance range will be evaluated for reasonableness.  
 Weights falling outside the range require that the balance be recalibrated using all S-class weights

Sheet No.: Balance - \_\_\_\_\_



ATTACHMENT 3

GRINDER CALIBRATION AND MAINTAINANCE LOG SHEET

Grinder # = \_\_\_\_\_

Sheet No.: Grinder - \_\_\_\_\_

**ATTACHMENT 4**

**VENTILATION HOOD CALIBRATION AND MAINTAINANCE LOG SHEET**

Ventilation Hood # = \_\_\_\_\_

Sheet No.: Hood - \_\_\_\_\_

**ATTACHMENT 5**

**HEPA VACUUM CALIBRATION AND MAINTAINANCE LOG SHEET**

Vacuum # = \_\_\_\_\_

Physically check the vacuum suction and note audible change in motor daily. If significant reduction in the vacuum suction or motor "strain" is audibly noted then the system check is unacceptable. If unacceptable, perform and document the HEPA filter and bag maintenance checks above and perform maintenance.

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**ATTACHMENT 6**

**OVEN CALIBRATION AND MAINTAINANCE LOG SHEET**

Oven # = \_\_\_\_\_

Sheet No.: Oven - \_\_\_\_\_